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Letter Report

Fabrication of Intermetallic Compounds by Solid State Reaction of Roll-Bonded Materials

Contract No. N00014-85-C-0857

Prepared for:

Office of Naval Research
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Quarterly Progress Report (#17)
(MCR-85-721)

1.0 **Contract Number:**
N00014-85-C-0857

2.0 **Reporting Period:**
8/10/90-11/9/90

3.0 **ONR Scientific Officer:**
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4.0 **Work Performed At:**
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5.0 **Principal Investigator:**
Dr. William C. Moshier

6.0 **Project Title:**
Fabrication of Intermetallic Compounds by Solid State Reaction of Roll-Bonded Materials

7.0 **Description of Research:**

7.1 Objectives of Present Research

- Intermetallic compounds offer very high specific material properties and property retention at elevated temperatures. However, reliable processing techniques to fabricate these materials have not been developed. This program will investigate the use of a novel, low cost fabrication technique involving a deformation-solid state reaction bonding process to form titanium beryllides. The objectives of this investigation are to:

  — Establish reaction kinetics for the formation of Ti beryllide intermetallic compounds,

  — Establish processing parameters needed to reproducibly fabricate these materials,

  — Characterize the properties of Ti beryllides fabricated using a deformation-solid state reaction bonding process,

  — Investigate the effects of alloying additions on the crystal structure of Ti beryllides and determine how these changes impact mechanical properties.

7.2 Summary of Work Accomplished During Previous Reporting Period

- Characterization and Testing of Ti Beryllide Foils

  Fabrication and room temperature mechanical testing of the sputtered foils were completed in the last quarter. Tensile fracture strength was measured for foils containing between 90.1 and 94.6 a/o Be, and was found to increased with Be concentration, as shown in Figure 1. Fracture surfaces on all of the samples were smooth at low magnifications. Observations at higher magnifications showed that the failure was transgranular and the features on the fracture surface change significantly with Be concentration, as shown in Figure 2. Fracture morphology appeared smoother and more cleavage-like as the Be concentration decreased, and the change in morphology correlated with the decrease in fracture strength.

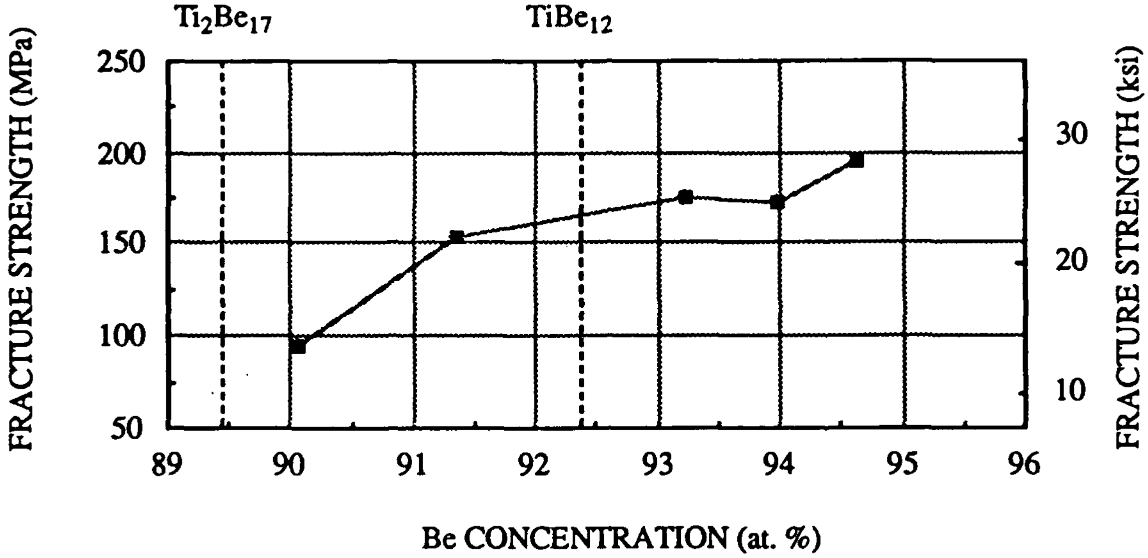

Figure 1. Fracture strength as a function of Be concentration indicates that the strength of the beryllide increases with Be concentration. Note that the actual structure at each Be concentration is $TiBe_{12}$.

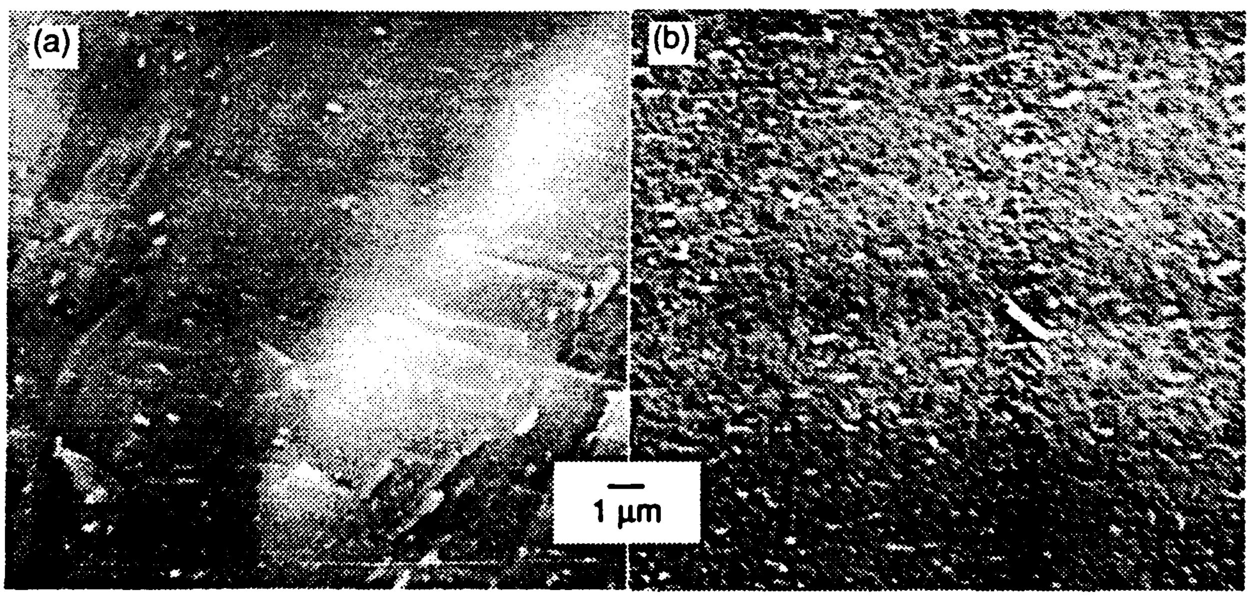

Figure 2. Fracture surface of Ti beryllide foil containing a) 90.05 a/o Be and b) 93.97 a/o Be. The smooth cleavage-type fracture of the sample containing 90.05 a/o Be corresponds with a lower fracture strength.

Room temperature mechanical properties for the sputtered beryllide foils were significantly higher than for other beryllides that had been previously tested. Tensile strength values reported in the literature for Nb_2Be_{17} were near 120 MPa, whereas the strength of the $TiBe_{12}$ foils ranged between 90 to 205 MPa, depending on the Be concentration. Our work demonstrated that reducing grain size and removing inclusions from the beryllides will result in improve properties.

7.3 Progress During the Reporting Period

- Interim Report

Interim Report, MCR-90-576, entitled, "Fabrication of Intermetallic Compounds Using a Solid State Reaction Bonding Process," was completed during this period and distributed to the appropriate U.S. Government agencies. Requests for this document should be submitted to the Office of Naval Research, Arlington, VA, 22217-5000.

- Microstructural Characterization of the Sputtered Foils

Results of this work to date have shown that the sputtered material had a grain size on the order of 200-300 nm. Although inclusions and particles were not found in the foil, the microstructure showed a high density of defects. Figure 3 shows a bright field image of a foil containing 93 a/o Be with submicron-sized grains, and the crystal structure is $TiBe_{12}$ as shown by the ring pattern in Figure 3b. At higher magnifications, Figure 4, the individual grains were readily seen, and the high density of defects present in the grains were locked in during the growth of the foil.

Annealing studies were performed on the foils in an effort to stress relieve the material. Foil shape was very sensitive to the heat treatment. Exposing the foil to temperatures between 750 and 1250°C in air for one hour resulted in curling of the foils, with increased deformation at higher the Be concentrations. Foil deformation at elevated temperatures interfered with the high temperature testing, since the foils would bend and crack during the heating cycle. To overcome this problem, the foils are being restrained with sheets of alumina during the heat treatment, which keeps them flat. Removal of residual stresses using this process has been successful for small pieces of material, and tests are now being conducted on larger sections. TEM of the annealed material will be performed once we have defined an optimum heat treatment cycle.

- Fabrication of Roll Bonded and Reacted Beryllides Foil

Fabrication of a roll bonded foil was completed in the past quarter, and heat treatment studies are now in progress to determine the optimum HIP'ing conditions and can material for the reaction bonding process. Material has been designed so that it can be completely reacted in less than eight hours.

7.4 Tasks for the Next Period

- Testing

Optimizing the process for reaction bonding the laminant will be completed early in the next quarter, and we will begin fabricating and testing this material to compare it with the sputtered foil. Testing will initially be conducted at room temperature. Once a suitable heat treatment has been established for both the sputtered foils and the reaction bonded material, high temperature testing will be performed in air.

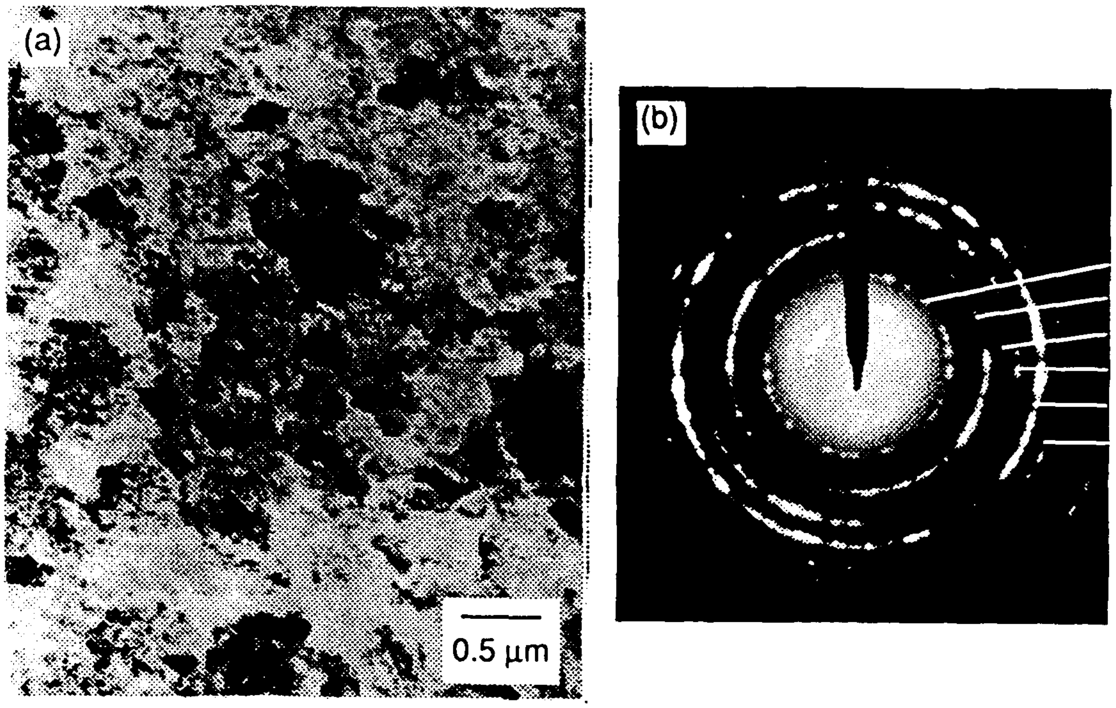

Figure 3. Microstructure of a foil containing 93 a/o Be shows a very fine grain size with no evidence of inclusions. Selected area diffraction of the foil shows only the presence of the $TiBe_{12}$ phase.

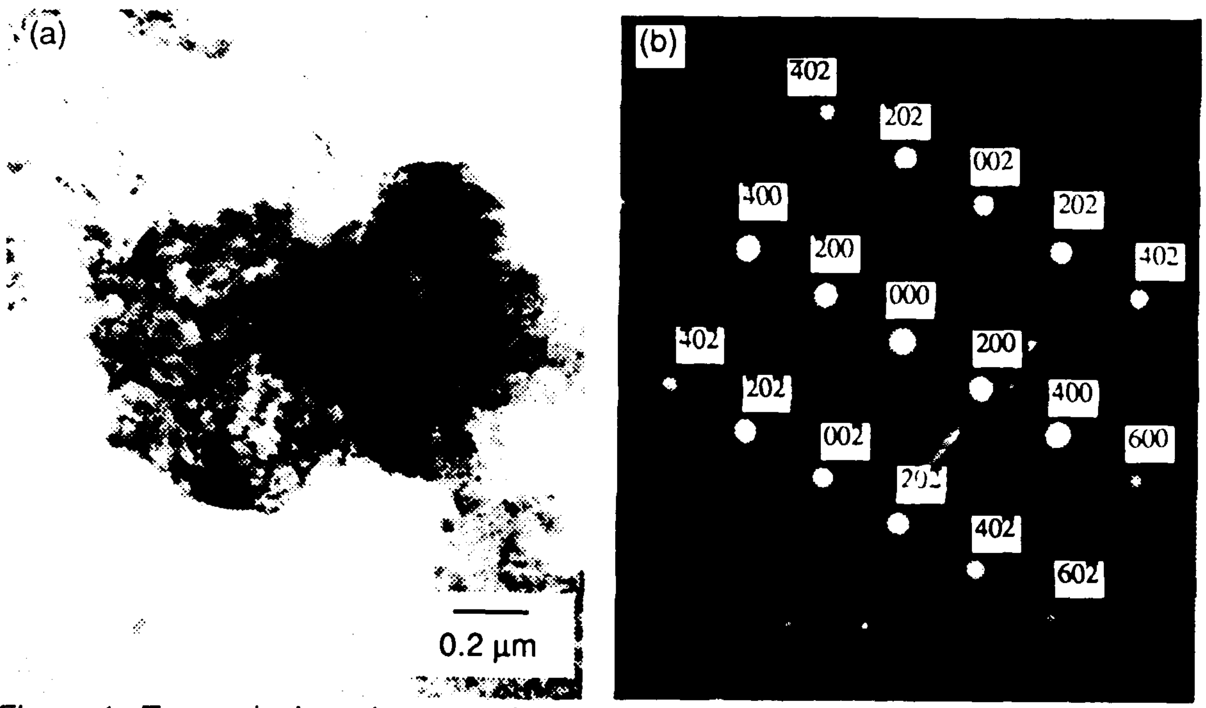

Figure 4. Transmission electron microscopy of titanium beryllide foil showing the: a) bright field image, and b) selected area diffraction pattern of the diffracting grain on the left indicting that these $TiBe_{12}$ grains grew along the (020) direction.

- TEM Analysis of Foils

TEM will continue on the sputtered foils to characterize their microstructure. Characterization of the roll bonded material will also be performed to determine the extent of reaction, reaction products present in the sheet, and the presence of defects in the microstructure.

7.5 Participants On The Program (Last Quarter)

Name	Task
Nuclear Metals Inc., Concord, MA	Extrusion & Specimen Preparation
EG&G, Rocky Flats, Golden, CO	Sputter-deposit Ti-Be foils
Manufacturing Sciences Corp, Oak Ridge, TN	Roll Bonding Ti-Be laminant
LANL, Los Alamos, NM	TEM of Ti beryllide foils